1013,257

PATENT SPECIFICATION

DRAWINGS ATTACHED

1013,257

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Int. Cl.:—C 22 c // B 23 p

COMPLETE SPECIFICATION

Improvements in or relating to Annealing

We, THE BRITISH IRON AND STEEL RESEARCH ASSOCIATION, a British Company of 24, Buckingham Gate, London, S.W.1, formerly of 11, Old Park Lane, London, W.1, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process for the annealing of cold worked steel in elongated form, that is in the form of sheet, strip or wire.

Steel sheet and strip is produced for the tinplate industry in a number of grades having different ranges of hardness and these grades of material are known in the British and American tinplate industries as "Temper 1," "Temper 2," etc. up to "Temper 6," the latter being the hardest and having a hardness of from 65 to 70 R30T units (84 to 86.5 RI5T units) in the "as annealed" conditions and of 68 to 73 R30T units (85.5 to 88.5 RI5T units) after temper rolling and ready for tinning (R30T and RI5T units are Rockwell hardness numbers using the superficial Rockwell Tester with a 30 Kg. and 15 Kg. load, respectively).

We have now developed an improved process of annealing cold worked low carbon steel in elongated form which enables material having any desired range of hardness to be produced, and which, in particular, enables Temper 6 material to be produced from low carbon steel. It is not possible to produce Temper 6 materials from low carbon steel by conventional annealing procedures, it being necessary to use rephosphorised steel containing approximately 0.1 to 0.14% phosphorus.

The process according to the invention comprises rapidly quenching the steel from an annealing temperature, quench ageing the steel and, before quench ageing is complete, rolling the steel and strain ageing it until a stable product is obtained. Since the strain ageing is effected before quench ageing is complete, the last stage of the process can be considered to be a combined quench ageing-strain ageing treatment.

The annealing temperature can be either sub-critical, i.e. a temperature below the Ac_1 transformation point at which ferrite transforms into austenite, but sufficiently high for recrystallisation of the cold worked ferrite to take place, or super-critical, i.e. at or above the Ac_1 transformation point, and up to the temperature at which all the ferrite becomes transformed into austenite. The actual limits of these ranges will depend upon the composition of the steel; in the case of the low carbon steels used in the production of tinplate, the minimum temperature at which recrystallisation of the cold worked ferrite takes place is about 680°C, the Ac_1 point is about 723°C., and all the ferrite is transformed to austenite at about 880°C. The overall range of suitable annealing temperatures for these steels is, therefore, from 680°C to 880°C, the range from 680°C to 723°C being the sub-critical range and the range from 723°C to 880°C being the range of suitable super-critical annealing temperatures.

Strain ageing has the effect of increasing the hardness of the steel and we have found that for any given strain ageing conditions, i.e. degree of reduction effected by rolling and temperature of strain ageing, the increase in hardness is substantially independent of the initial hardness of the steel. That is to say substantially the same increase in hardness is

imparted to the steel by strain ageing under given conditions whatever the hardness of the steel prior to strain ageing.

Steels which have been quenched from an annealing temperature mentioned above to a temperature somewhat above the Ac_1 point, i.e. in the case of tinplate steels, from an annealing temperature of from 680°C to 780°C, exhibit a quench ageing effect in that there is a progressive decrease in the as-quenched hardness with an increasing period of quench ageing; at 200°C quench ageing is completed in about 15 minutes typically giving a decrease in hardness of about 1.5 RI5T units. When annealing is effected within the temperature range just mentioned, therefore, the period for which the steel is quench aged prior to rolling affords a useful means of controlling and varying the final hardness of the product. The hardest products, other variables being the same, are therefore obtained by omitting the quench ageing step and rolling immediately after quenching. Quench ageing for a short period, that is, for example, by effecting rolling within about 1 minute of quenching, does not greatly reduce the as-quenched hardness and this degree of quench ageing is usefully employed.

Steels which have been quenched from an annealing temperature of from about 780°C to the maximum annealing temperature mentioned above, do not exhibit a quench ageing effect and we have found that there is substantially no change in their hardness even when quench aged for several hours. There is, therefore, no advantage to be gained by quench ageing steels which have been annealed at these temperatures and, indeed, any attempt to quench age under these circumstances is clearly economically undesirable since quench ageing necessarily involves holding the steel at an elevated temperature. Where reference is made in this specification to "quench ageing" a steel annealed at a temperature above 780°C, it is to be understood that this refers to holding the steel at a suitable temperature for quench ageing and does not mean that any ageing effect as evidenced by variation in hardness takes place.

Quench ageing (if effected) and strain ageing may be carried out at a temperature of from 100 to 250°C and the temperature employed is one of the factors affecting the hardness of the final product. In principle, the lower the temperature within the specified range, the harder the product, but at the same time, the lower the temperature, the longer will it take to complete the quench ageing-strain ageing. For these reasons it is preferred to use temperatures in the range 100° to 200°C, and advantageously of from 150° to 160°C.

The reduction effected by the rolling step can generally be about the same as that effected in temper rolling annealed material in conventional processes and normally need not be more than 3%. At this level an increase in the amount of reduction has little effect on the final hardness of the material, but there is a significant increase in hardness when the amount of reduction is raised from, say, 0.7 to 1.7%.

Within the general category of low carbon steels such as are used for the production of tinplate, there are variations in carbon content and the latter has a significant effect on the final hardness of the product, the higher the carbon content, other variables being the same, the harder the product. The hardness of the product is also directly related to the annealing temperature, the higher the latter, the harder being the product. By using the optimum conditions of the process for obtaining hardness, material meeting Temper 6 requirements can be obtained from steel containing at least 0.1% carbon using sub-critical annealing temperatures, but even with the use of these optimum conditions, Temper 6 material cannot be obtained from steel containing less than 0.1% carbon if a sub-critical annealing temperature is used. Temper 6 material can, however, be obtained from a steel containing less than 0.1% carbon if a super-critical annealing temperature is used.

In the commercial production of tinplate and galvanised steel, the strain ageing step of the present process is preferably carried out concurrently with (and as a result of) a step in the subsequent treatment of the steel in which the latter is heated; in the production of tinplate this may be the flow brightening or lacquering treatment and in the production of galvanised steel, the galvanising treatment, all of which treatments raise the steel to a suitable temperature for strain ageing. When the annealing temperature employed is above about 780°C (so that there is no quench ageing effect), the process according to the invention preferably takes the form of quenching from the annealing temperature, advantageously into water, temper rolling the quenched material either immediately or some time after quenching depending upon whether or not annealing is carried out in line with the after treatments and then passing the temper rolled material to the plating (hot dip or electrolytic) line or galvanising line.

An increase in the hardness of the steel can be obtained by adding a further temper rolling step or a further rolling and strain ageing step to the process, this additional step being carried out after completion of the combined quench ageing-strain ageing treatment. Where an additional temper rolling step is used, this may be similar to the temper rolling carried out in the conventional production of tinplate, i.e. it may consist of a cold reduction of up to 3%, while an additional strain ageing treatment will be generally similar to the first strain ageing treatment. The time required to complete the combined

quench ageing-strain ageing treatment and the second strain ageing treatment, when the latter is used, will as indicated above, depend upon the ageing temperature; at the preferred temperature of from 150° to 160°C, ageing will normally be complete and a stable product obtained, in about 45 minutes. In order to minimise heat loss from the steel during ageing, it is preferably coiled if in the form of strip or wire, or stacked if in the form of sheet, preferably in a heat insulated enclosure or jacket.

The process is readily adapted for continuous operation for the treatment of material in continuous form, i.e. strip or wire, the steel being passed through a heating zone in which it is rapidly heated up to the annealing temperature, preferably in less than 10 seconds, and then immediately or after a short holding period, preferably less than 5 seconds, passed into a quenching medium maintained at or slightly above the ageing temperature. After quenching the steel is rolled in-line, preferably within 1 minute, and advantageously within 5 seconds, of quenching and then coiled at the rolling temperature. After holding in coil for, say, 45 minutes, the steel is ready for tinning. If a further increase in hardness is required, a second rolling treatment can be applied before tinning. The actual tinning operation or the flow-brightening step in electro tinplate production strain ages the material adequately after the rolling. Apart from the ability to produce the harder tinplate grades, more particularly Temper 6, this process offers considerable advantages over conventional continuous annealing cycles as regards the time (and therefore the space) required for the in-line treatments. Thus conventional continuous annealing cycles involve a relatively long cooling period from the annealing temperature (typically from 90 to 120 seconds), whereas the whole of the in-line treatments of the present process need take no longer than 30 seconds (i.e. 10 seconds for heating, 5 seconds hold at annealing temperature, 5 seconds for quenching, 5 seconds for quench ageing, and 5 seconds for rolling.)

In order that the invention may be more fully understood, the following examples are given by way of illustration only.

EXAMPLE 1.

Samples of three steels have the following analyses were treated:

Carbon Content	Mn	P	S	Ni	Cu	Sn
0.07	0.28	0.008	0.016	0.005	0.07	0.01
0.10	0.40	0.014	0.020	0.05	0.05	0.01
0.14	0.42	0.022	0.026	0.06	0.07	0.01

In each case, the steel was in the hard condition having been cold rolled nominally 80% to 0.013 inch thickness.

The samples were heated to 700°C in a salt bath, immediately quenched into oil at 160°C and held for 4 seconds before water quenching. The samples were then cleaned and reheated to 160°C and quench aged for 0, $\frac{1}{4}$, $\frac{1}{2}$, 15 and 60 minutes at this temperature prior to rolling to 1.7% extension. After rolling, the samples were aged at 160°C.

The variations in hardness with ageing time after rolling at different stages in the quench ageing process are shown in Figures 1, 2 and 3 of the accompanying drawings which are curves obtained by plotting the hardness (in R30T units) against ageing time in minutes on a logarithmic scale. Figure 1 illustrating the variations from the 0.07% carbon steel, Figure 2 those for the 0.010% carbon steel and Figure 3 those for the 0.15% carbon steel. The Erichsen bend values for these steels in the fully strain aged condition, after various quench ageing periods prior to rolling, are given in the following Table.

Treatment	Erichsen Values		
	0.07% C	0.10% C	0.14% C
As quenched roll, no S.A.	7.8	6.5	7.9
As quenched roll, 1 hr. S.A.	7.3	6.0	5.7
¼ min. Q.A., roll, 1 hr. S.A.	—	5.9	6.3
½ min. Q.A., roll, 1 hr. S.A.	—	6.0	6.1
15 min. Q.A., roll, 1 hr. S.A.	—	6.6	5.7
60 min. Q.A., roll, 1 hr. S.A.	6.8	6.5	6.3
60 min. Q.A., roll, no S.A.	7.7	7.3	7.8

Q.A. = Quench Aging; S.A. = Strain Ageing.

These results show that when using a subcritical annealing temperature, while Temper 6 requirements were not obtained with any of the treatment conditions used in the case of the 0.07% carbon steel, Temper 6 requirements were obtained in the case of the 0.10% carbon steel with quench ageing times of up to ½ minute prior to rolling and in the case of the 0.14% carbon steel were obtained with quench ageing times of up to 60 minutes prior to rolling.

EXAMPLE 2

Samples of the same steels as were used in Example 1 were heated to 700°C and quenched as before and were rolled to 1.7% reduction in the as quenched condition, fully aged at 160°C, then given a further 0.7% temper roll reduction and strain aged at 160°C. The variation in hardness during the second ageing treatment are shown in Figure 4 of the accompanying drawings which shows curves obtained by plotting hardness (in R30T units) against ageing time in minutes on a logarithmic scale, for each of the three steels. The final hardnesses of the fully strain aged products were 72.5 (0.14% carbon steel), 72.2 (0.10% carbon steel) and 70.0 (0.07% carbon steel); the corresponding Erichsen values were 6.1, 6.4 and 6.2.

These results show that material meeting Temper 6 requirements can be obtained even with the relatively very low carbon content of temperature by the use of a second strain ageing treatment.

EXAMPLE 3.

Steel strip having the following analysis was treated: 0.08% C; 0.035% S; 0.013% P; 0.34 Mn; 0.05% Ni; 0.06% Cu; 0.01% Sn. The strip was in the hard condition having been cold rolled nominally 85% to 0.006 inch thickness.

The strip was passed through a continuous annealing line at a constant strip speed of 100 ft/min; in this line it was heated to the annealing temperature in about 12 seconds and then immediately passed into a molten lead-bismuth bath where it was quenched to 200°C. Following removal from the lead-bismuth bath, the strip was quenched into water. Four runs were made using different annealing temperatures, viz. 700°C, 740°C, 800°C and 850°C.

A part of the strip from each run was temper rolled immediately following water quenching to obtain a 1.7% reduction using 6½ inch diameter smooth rolls, heated to 200°C and strain aged in coil at this temperature for 100 minutes; the hardness of the steel was determined at the beginning, during and at the end of this period (all hardness measurements were in RI5T units, diamond anvil).

Another part of the strip from each run was quench aged for 1 hour at 200°C following water quenching and was then temper rolled and strain aged in the same way as the first part.

The results obtained are shown in Figure 5 of the accompanying drawings which shows curves obtained by plotting hardness (in RI5T units) against strain ageing time in minutes on logarithmic scale, two curves being shown for the steel annealed at each of the four annealing temperatures.

These curves show (a) that there is a progressive increase in final hardness with annealing temperature, (b) that quench ageing for 1 hour has substantially no effect on the final hardness when annealing temperature of 800° and 850°C are used, whereas it does reduce the final hardness when annealing is effected at 700°C and 740°C, and (c) that substantially the same increment in hardness is obtained with a given strain ageing treatment

whatever the hardness of the steel at the commencement of the strain ageing treatment.

As a confirmation of point (b) above, further samples of the same steel were annealed at 800°C and 850°C as described above and, after water quenching, were quenched aged at 200°C for several hours. No decrease in hardness was observed.

WHAT WE CLAIM IS:—

1. A process for annealing cold worked low carbon steel in elongated form, which comprises rapidly quenching the steel from a subcritical annealing temperature, quench ageing the steel and, before quench ageing is complete, rolling the steel and strain ageing it until a stable product is obtained.

2. A process according to claim 1, in which quench ageing and strain ageing is effected at a temperature of from 100° to 200°C.

3. A process according to claim 2, in which quench ageing and strain ageing is effected at a temperature of from 150° to 160°C.

4. A process according to any of claims 1 to 3, in which the steel is rapidly quenched to a temperature at which quench ageing takes place.

5. A process according to any of claims 1 to 4, in which rolling is carried out within 1 minute of quenching.

6. A modification of the process according to any of claims 1 to 5, in which the steel is rolled immediately after quenching.

7. A process according to any of claims 1 to 6, in which the steel is rolled to obtain a reduction of approximately 1.7%.

8. A process according to any of claims 1 to 7, in which the steel is subjected to further rolling after completion of the strain ageing.

9. A process according to any of claims 1 to 7, in which the steel is subjected to further rolling and strain ageing after completion of the first strain ageing.

10. A process according to claim 9, in which the further strain ageing is effected at a temperature of from 100° to 200°C.

11. A process according to claim 10, in which the further strain ageing is effected at a temperature of from 150° to 160°C.

12. A process according to any of claims 8 to 11, in which the further rolling is effected to obtain a reduction of up to 3%.

13. A modification of the process according to any of the preceding claims, in which the steel is rapidly quenched from a temperature above its Ac_1 point and below the temperature at which all the ferrite is transformed into austenite.

14. A process for annealing cold worked low carbon steel in elongated form, which comprises rapidly quenching the steel from an annealing temperature above about 780°C into water, temper rolling the quenched steel and then strain ageing the rolled steel to obtain a stable product in a subsequent heat treatment of the steel.

15. A process for annealing cold worked low carbon steel substantially as herein described in Example 1 or 2.

16. A process for annealing cold worked low carbon steel substantially as herein described in Example 3.

17. Low carbon steel which has been annealed by the process claimed in any of the preceding claims.

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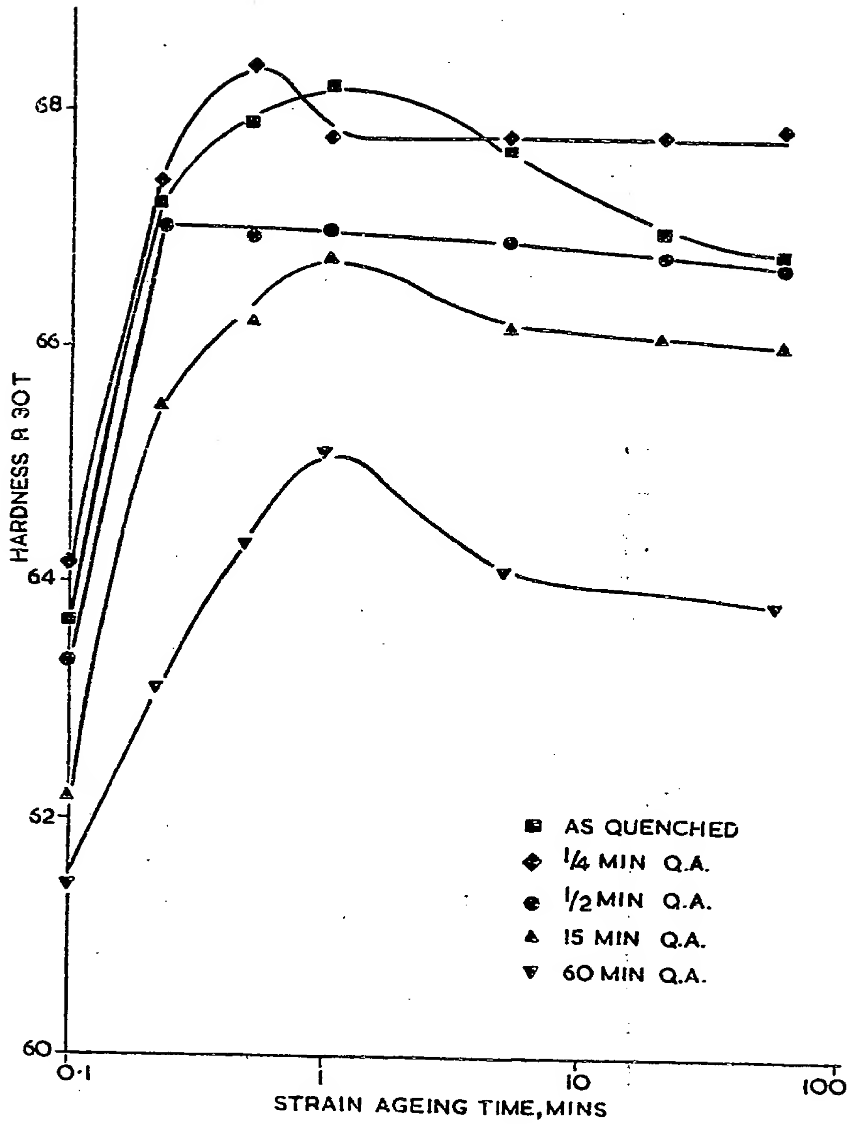
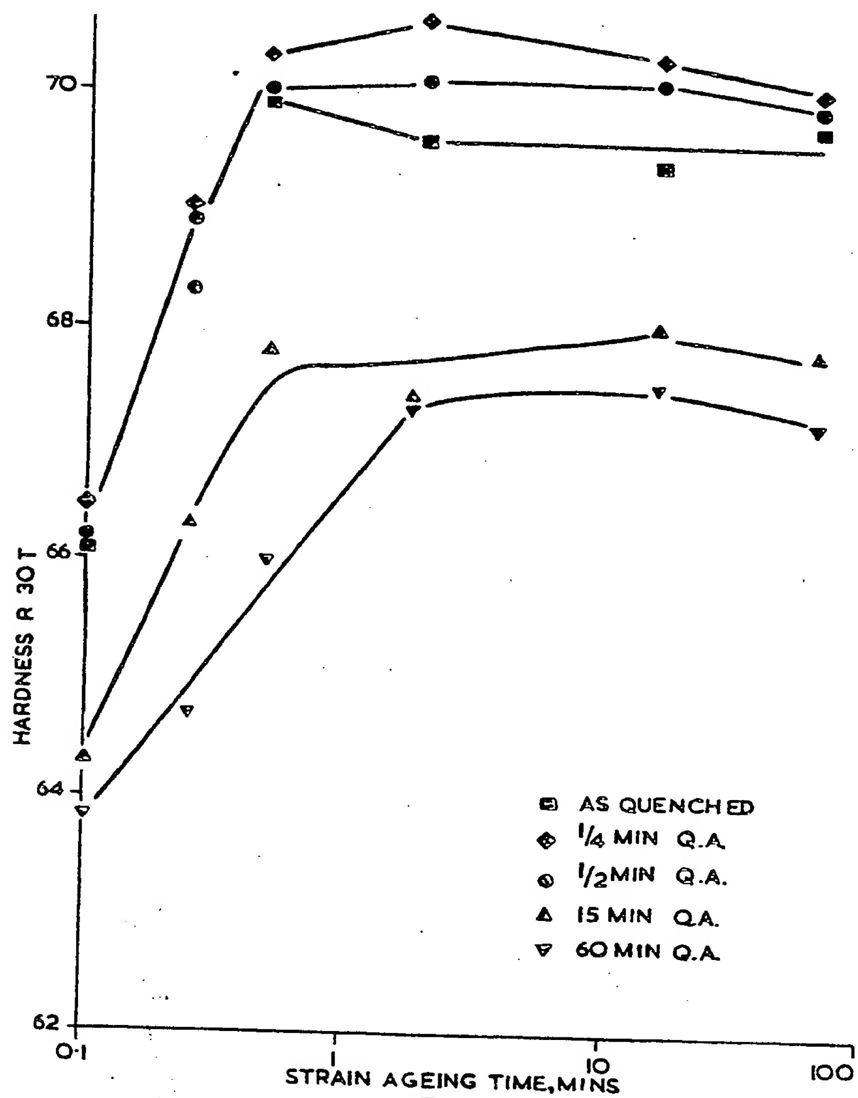

68
66
64
62
60
HARDNESS R 30T
0·1
1
10
100
STRAIN AGEING TIME, MINS
AS QUENCHED
1/4 MIN Q.A.
1/2 MIN Q.A.
15 MIN Q.A.
60 MIN Q.A.

FIG.1.



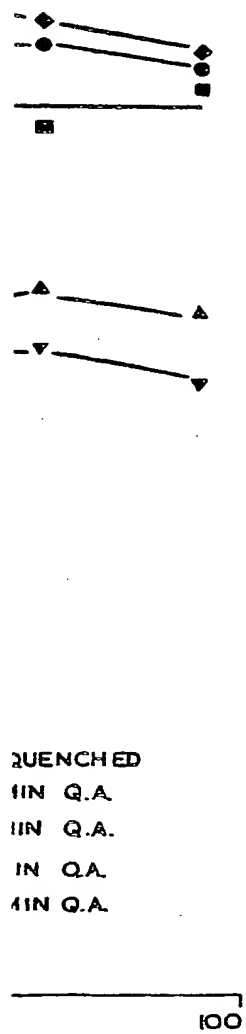
70
68
66
64
62
HARDNESS R 30 T
0·1
1
10
100
STRAIN AGEING TIME, MINS
AS QUENCHED
1/4 MIN Q.A.
1/2 MIN Q.A.
15 MIN Q.A.
60 MIN Q.A.

FIG.2.

1013257 COMPLETE SPECIFICATION

5 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheets 2 & 3



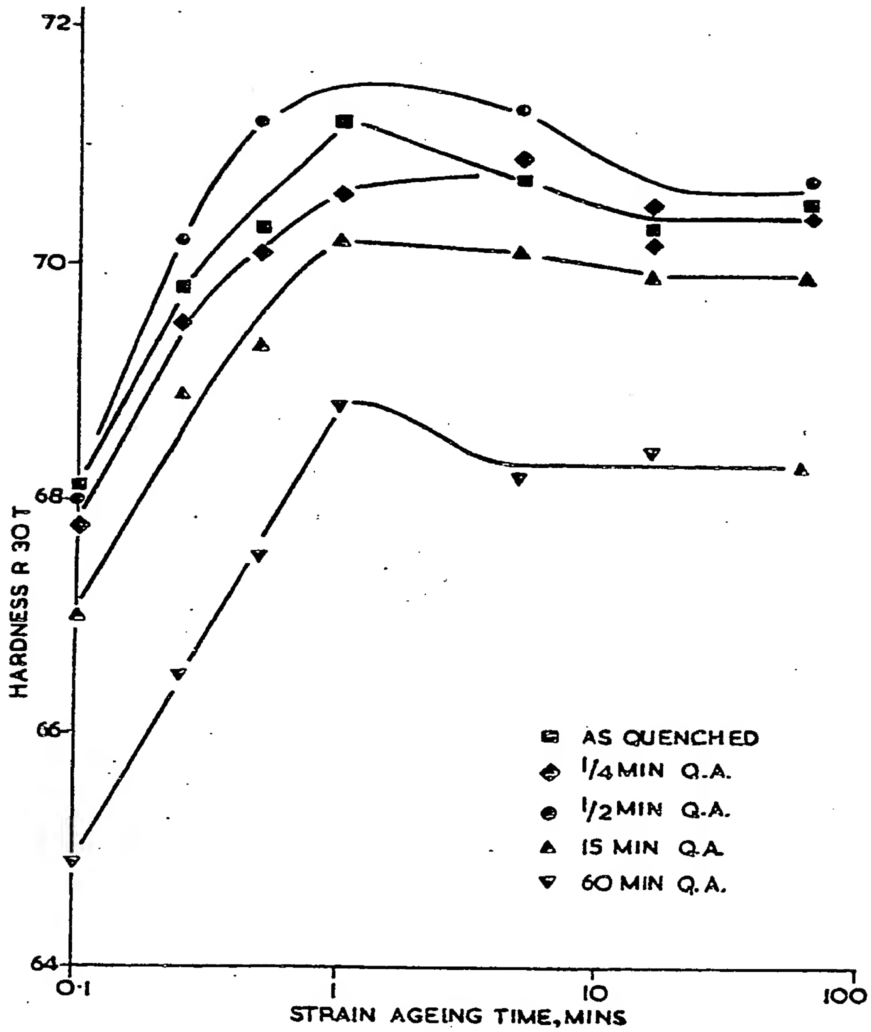


FIG.3.

1013257 COMPLETE SPECIFICATION

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Sheets 2 & 3

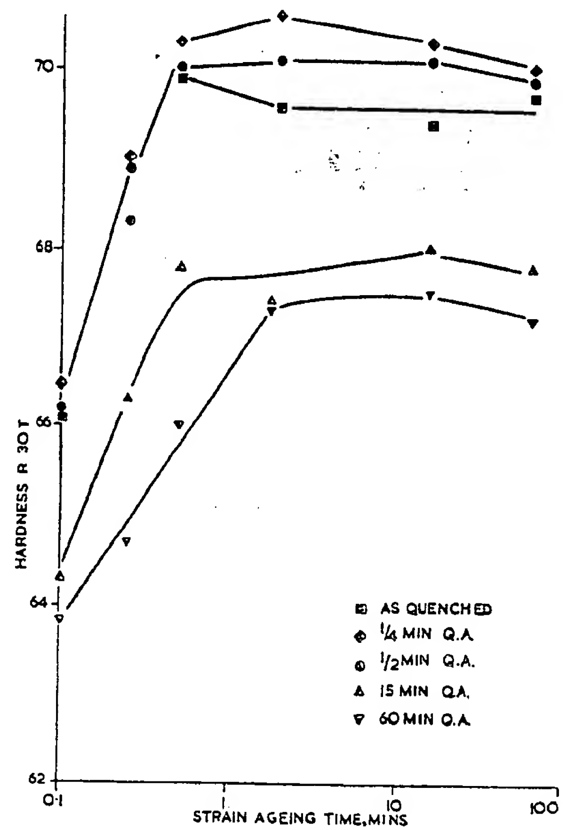


FIG. 2.

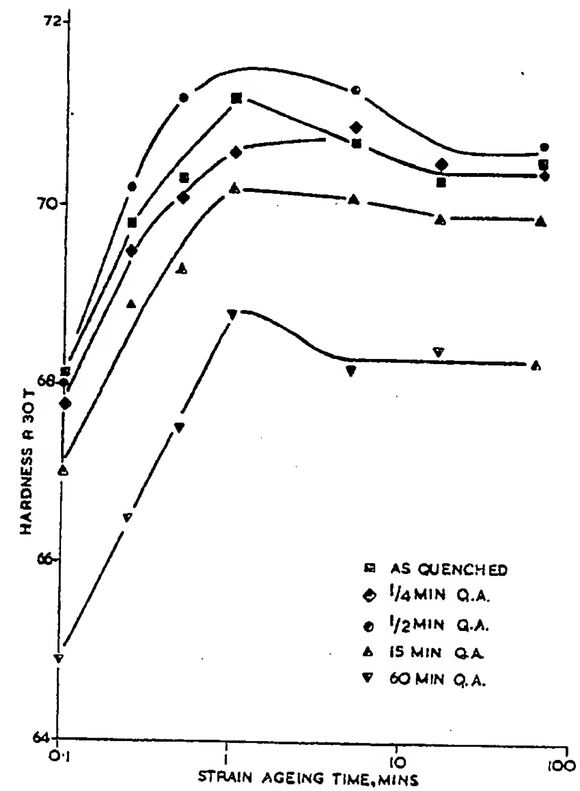


FIG. 3.

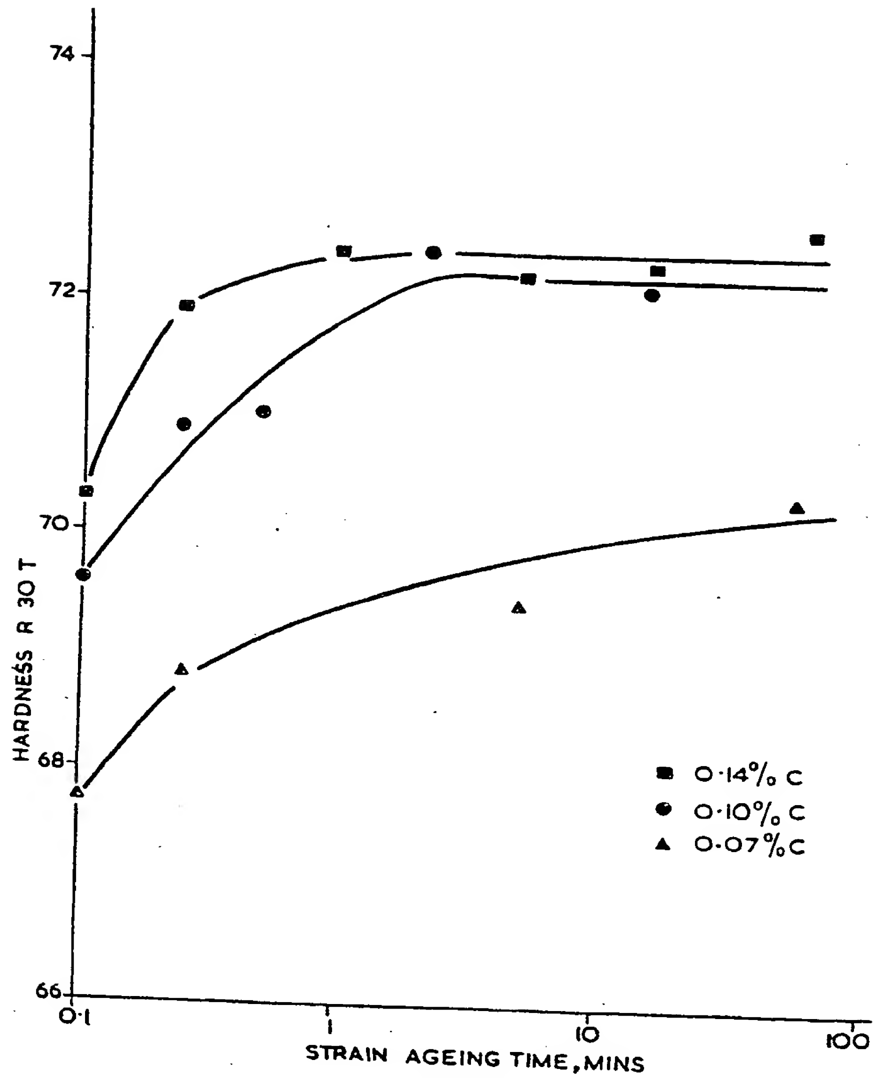
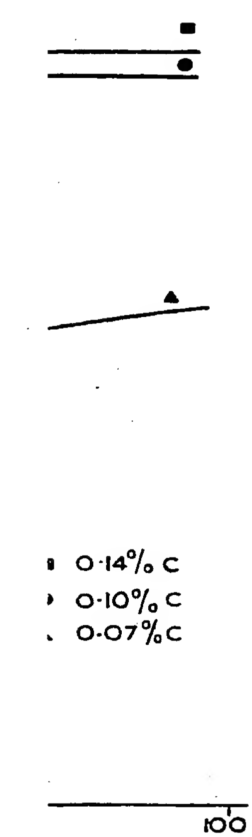

74
72
70
68
66
HARDNESS R 30 T
0·1
1
10
100
STRAIN AGEING TIME, MINS
0·14% C
0·10% C
0·07% C

FIG.4.

1013257 COMPLETE SPECIFICATION

5 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheets 4 & 5



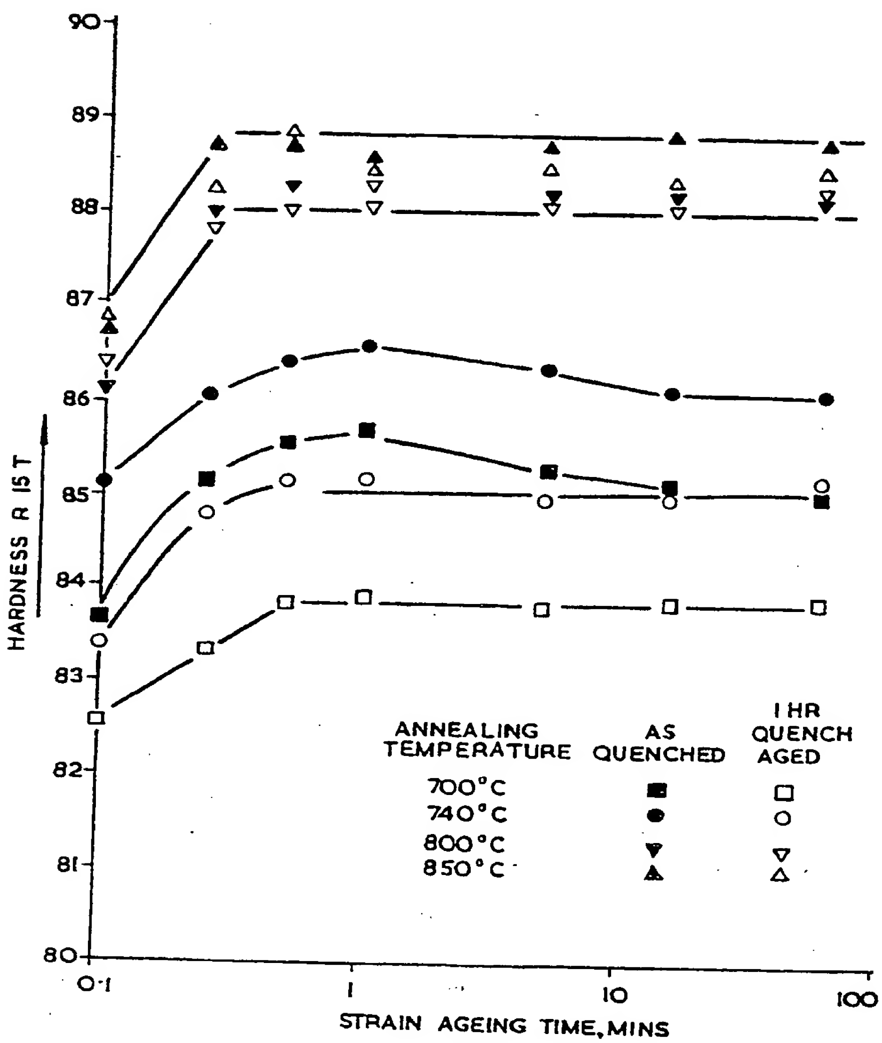


FIG.5.

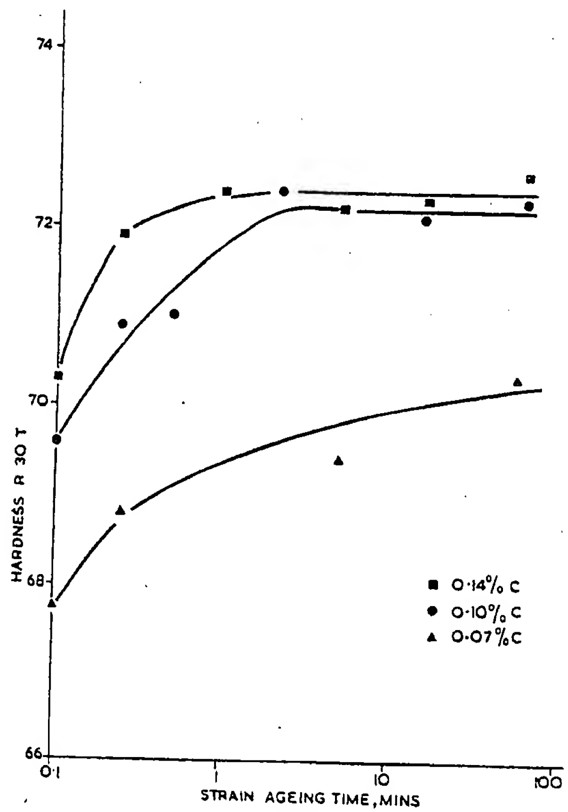

HARDNESS R 30 T
74
72
70
68
66
0·1
1
10
100
STRAIN AGEING TIME, MINS
■ 0·14% C
● 0·10% C
▲ 0·07% C

FIG.4.

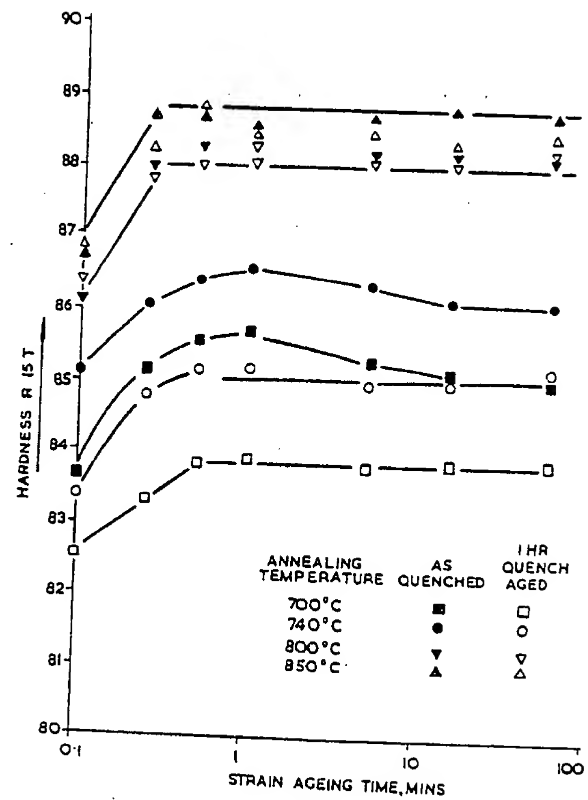

HARDNESS R 15 T
90
89
88
87
86
85
84
83
82
81
80
0·1
1
10
100
STRAIN AGEING TIME, MINS
ANNEALING TEMPERATURE
AS QUENCHED
1 HR QUENCH AGED
700°C
740°C
800°C
850°C

FIG.5.